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PATENT

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APPLICATION

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FOR

UNITED STATES LETTERS PATENT

FOR

SPUNLACED POLY(VINYL ALCOHOL) FABRICS

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BY

Feng Qin, a citizen of the Peoples Republic of China, residing at 3232F

Windscape Village, Norcross, Georgia 30093, U.S.A.; **Youzhen Ding**, a citizen of the
Peoples Republic of China, residing at 5632 Hammond Dr. Norcross, Georgia 30071

25 **Dong Dai**, a citizen of the Peoples Republic of China, residing at 3125 Paces
Woods Drive, Lawrenceville, Georgia 30244 U.S.A.; and **Baosheng Lee**, a citizen of
Taiwan, Republic of China, residing at 3990 Bridlewood Drive, Duluth, Georgia,
30136, U.S.A.

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SPUNLACED POLY(VINYL ALCOHOL) FABRICS

FIELD OF THE INVENTION

The present invention relates to nonwoven fabrics made from poly(vinyl alcohol), and particularly to nonwoven fabrics made from poly(vinyl alcohol) by the spunlace process.

BACKGROUND OF THE INVENTION

Spunlaced fabrics are produced by carding a plurality of fabrics into a sheet, and subsequently passing the sheet under water jets to hydroentangle the fibers. Spunlaced fabrics are nonwoven, and thus do not require complex weaving steps or machinery for their preparation. Spunlaced fabrics also are different from other nonwoven fabrics, which must be thermobonded, chemically bonded, or stitchbonded, to produce a fabric of sufficient strength for commercial use.

U.S. Patent No. 5,093,190 to Kwok et al. (incorporated herein by reference) discloses a process for making spunlaced acrylic/polyester fabrics. Johnson & Johnson and Maxxim Medical manufacture and sell spunlaced fabrics made from polyester and cellulose fibers. The above mentioned spunlaced fabrics are feasible for many commercial applications. However, it would be desirable to improve the performance of these fabrics in a number of respects, including bursting strength, air permeability, tensile strength, flammability, absorbency, impact penetration, water vapor transmission, and water repellency.

SUMMARY OF THE INVENTION

It has been discovered that poly(vinyl alcohol) spunlaced fabrics have improved physical properties over the spunlaced fabrics of the prior art. Thus, in one respect the invention provides a poly(vinyl alcohol) fabric produced by a method comprising the consecutive steps of supporting a plurality of poly(vinyl alcohol) fibers on a mesh screen to form a web, pressure liquid entangling the web, and drying the web.

In another aspect the invention provides a fabric comprising a poly(vinyl alcohol) fibrous web, wherein the fabric is nonwoven, binding adhesives are substantially absent from the fabric, heat fusion is substantially absent from the fabric, needlepunching is substantially absent from the fabric, and stitchbonding is substantially absent from the fabric.

In yet another aspect the invention provides a method of finishing a poly(vinyl alcohol) fabric to impart water repellence to the fabric comprising contacting the fabric with an aqueous finishing formulation, and subsequently drying the fabric and/or curing the finish at a temperature above the water solubility temperature of the poly(vinyl alcohol).

In still another embodiment the invention provides a method of making a poly(vinyl alcohol) fabric comprising (a) supporting a plurality of poly(vinyl alcohol) fibers on a mesh screen to form a web; (b) pressure liquid entangling the web; and (c) drying the web.

Additional aspects and advantages of the invention will be set forth in part in the description which follows, and in part will be obvious from the description, or may be learned by practice of the invention. The advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed.

DISCUSSION OF THE INVENTION

The present invention may be understood more readily by reference to the following detailed description of preferred embodiments of the invention and the Examples included therein.

Before the present materials and methods are disclosed and described, it is to be understood that this invention is not limited to specific methods or materials as such may, of course, vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting.

Use of Terms

As used in the specification and the appended claims, the singular forms "a," "an" and "the" include plural referents unless the context clearly dictates otherwise. Thus, for example, reference to "a fiber" includes mixtures of fibers.

Ranges are often expressed herein as from "about" one particular value, and/or to "about" another particular value. When such a range is expressed, another embodiment includes from the one particular value and/or to the other particular value.

Similarly, when values are expressed as approximations, by use of the antecedent "about," it will be understood that the particular value forms another embodiment.

Similarly, when ranges extend from one endpoint to another endpoint, another embodiment includes the range between the endpoints and excluding the endpoints.

- 5 References in the specification and concluding claims to parts by weight, of a particular element or component in a composition or article, denotes the weight relationship between the element or component and any other elements or components in the composition or article for which a part by weight is expressed. Thus, in a compound containing 2 parts by weight of component X and 5 parts by weight
10 component Y, X and Y are present at a weight ratio of 2:5, and are present in such ratio regardless of whether additional components are contained in the compound.

A weight percent of a component, unless specifically stated to the contrary, is based on the total weight of the formulation or composition in which the component is included.

- 15 A residue of a chemical species, as used in the specification and concluding claims, refers to the moiety that is the resulting product of the chemical species in a particular reaction scheme or subsequent formulation or chemical product, regardless of whether the moiety is actually obtained from the chemical species. Thus, an ethylene glycol residue in a polyester refers to one or more -OCH₂CH₂O- units in the polyester,
20 regardless of whether ethylene glycol was used to prepare the polyester. Similarly, a sebacic acid residue in a polyester refers to one or more -CO(CH₂)₈CO- moieties in the polyester, regardless of whether the residue is obtained by reacting sebacic acid or an ester thereof to obtain the polyester.

- By the term "effective amount" of a compound or property as provided herein is
25 meant such amount as is capable of performing the function of the compound or property for which an effective amount is expressed. As will be pointed out below, the exact amount required will vary from process to process, depending on recognized variables such as the compounds employed and the processing conditions observed. Thus, it is not possible to specify an exact "effective amount." However, an appropriate
30 effective amount may be determined by one of ordinary skill in the art using only routine experimentation.

Heat fusion refers to any method in which fibers are bonded by application of heat, and includes thermobonding.

Pressure liquid entangling refers to a process for entangling fibers in a web of fabric by spraying a plurality of liquid jets onto the web and thereby entangling the fibers. Pressure liquid entangling thus includes hydroentangling using jets of water.

Degree of hydrolysis also includes degree of saponification where saponification is employed in the preparation of poly(vinyl alcohol).

Description of the Invention

In one aspect the invention provides a poly(vinyl alcohol) fabric produced by a method comprising the consecutive steps of (a) supporting a plurality of poly(vinyl alcohol) fibers on a mesh screen to form a web; (b) pressure liquid entangling the web; and (c) drying the web.

The invention is preferably practiced with a poly(vinyl alcohol) and poly(vinyl alcohol) fibers meeting the following characteristics:

- 15 1. Degree of polymerization of poly(vinyl alcohol): Preferably from about 300 to about 5000, more preferably from about 800 to about 3000, and still more preferably from about 1200 to about 2000.
- 20 2. Degree of hydrolysis of poly(vinyl alcohol): Preferably greater than 80%, more preferably greater than 85%, even more preferably greater than 90%, still even more preferably greater than 95%, even further preferably greater than 97%, and still even further preferably greater than 98%.
- 25 3. Average denier of fibers: Preferably from about 0.1 to about 10, more preferably from about 0.5 and about 5, and even more preferably from about 1 to about 3 denier.
- 30 4. Average length of fibers: Preferably from about 4 mm to about 300 mm, more preferably from about 20 to about 100 mm, even more preferably from about 30 to about 60 mm, and most preferably about 38 mm.
5. Temperature above which the fiber is soluble, and below which the fiber is insoluble, in separate embodiments: 40 °C, 50 °C, 60 °C, 70 °C, 80 °C, 90 °C, 100 °C, and 110 °C. Alternatively, the fibers can be cold water soluble, or soluble at room temperature, to facilitate eventual disposal.

The pressure liquid entangling can be performed under any conditions that does not detrimentally affect the properties of the web in a substantial way. For hot water soluble poly(vinyl alcohol) the liquid entangling is preferably performed with water. Poly(vinyl alcohol) fibers that are cold water soluble or soluble at room temperature, 5 and which might be at least partially dissolved by hydroentangling, can be entangled by liquids which are nonexplosive, have a low boiling point, and do not readily dissolve poly(vinyl alcohol).

The pressure liquid entangling is preferably performed at a water pressure of from about 10 to about 200 bar, more preferably at a water pressure of from about 20 to 10 about 120 bar, and even more preferably at a water pressure of from about 40 to about 100 bar. The drying is preferably performed at a temperature of from about 20°C to about 230°C, more preferably from about 60°C to about 130°C, and even potentially at a temperature that exceeds the solubility temperature of the poly(vinyl alcohol). The drying is preferably performed by passing heated air through the web. Even more 15 preferably, the web is dried by passing it over a perforated drum that draws air through the fabric and into the perforated drum.

The method might also preferably comprise other steps, including, after step (a), the steps of cross-lapping the web; and stretching the web in the machine direction. The method might also comprise, after step (c), winding the web onto a roll.

20 The web preferably satisfies the following criteria after step (c):

1. Thickness: Preferably from about 0.05 to about 2 mm, more preferably from about 0.1 mm to about 1 mm, still more preferably from about 0.3 mm to about 0.6 mm, and most preferably about 0.4 mm.
2. Base weight (per 0.4 mm of thickness): Preferably from about 20 g/m² to about 25 400 g/m², more preferably from about 35 to about 200 g/m², even more preferably from about 40 to about 100 g/m², still even more preferably from about 50 to about 80 g/m², and most preferably about 70 g/m².

The poly(vinyl alcohol) fibers may also be carded along with other fibers selected from the group consisting of polyester, polypropylene, polyethylene, rayon, 30 cellulose, nylon, ethylene/(meth)acrylic acid copolymer, and other fibrous polymers known in the art. In addition, the method may further comprise, after step c, adhering a substantially impermeable layer to the web. The layer preferably comprises poly(vinyl

alcohol) polyethylene, polypropylene, polyester, ethylene/(meth)acrylic acid copolyester, or any other polymer known to form impermeable layers. The substantially impermeable layer can be adhered to the web by methods including extrusion coating, laminating, spraying, dipping and roll coating.

5 The finished web may further comprise, and preferably be substantially saturated with, a solvent, so that the web can be used as a wipe. Thus, the method can further comprise, after step (c), contacting (and optionally saturating to greater than 10%, 25%, 40%, 60%, 75%, 90%, or 100% saturation) the web with a solvent liquid such as isopropyl alcohol, water, methyl ethyl ketone, methyl propyl ketone, and
10 acetone.

The fabric might also be treated to impart water repellency. Thus, in still another embodiment the method further comprises contacting the web with an aqueous finishing formulation to impart water repellency to the fabric, preferably before step (c). One or both sides can be contacted. In one embodiment the aqueous finishing
15 formulation comprises a fluorocarbon and a wax and preferably contributes from about 0.01 to about 3 wt. % fluorocarbon, and from about 0.01 to about 15 or 20 wt. % wax, to the weight of the fabric.

An important attribute of the fabrics made by the process of this invention is their superior physical properties. Thus, the fabric preferably satisfies one or more of
20 the following properties, and can satisfy any combination of the following properties. These properties are especially useful in fabrics that are about 0.4 mm thick, and that have a base weight of about 70 g/m². It will be understood that the strength attributes given below can be extrapolated based upon increases or decreases from a 0.4 mm thick fabric having a base weight of about 70 g/m².

- 25 1. The fabric preferably has a tensile strength in the machine direction greater than about 13 pounds, more preferably about 17 pounds, and more preferably about 20 pounds, when measured for a one inch strip according to ASTM D5035-95.
2. The fabric preferably has a tensile strength in the cross direction greater than 13 pounds, more preferably about 17 pounds, and more preferably about 20 pounds, when
30 measured for a one inch strip according to ASTM D5035-95.
3. The fabric preferably has a bursting strength greater than about 50, 60, 70, or 80 psi when measured by ASTM D3776-96. *378681*

4. The fabric preferably has an air permeability of greater than about 100, 125, or 150 CFM/sq. ft. when measured by ASTM D737-96.
5. The fabric preferably has a flammability rating of IBE or DNI when measured according to ASTM D1230-94. This flammability rating is especially useful for surgical fabrics such as gowns and drapes that are exposed to laser surgery, and which are at greater risk of flammability.
6. The fabric preferably has a water impact penetration less than 1.5, 1.2, or 1.0 grams when measured by AATCC 42-94.
7. The fabric preferably has cumulative linting, when measured by INDA IST, of less than 7000, 5000, or 4000 in the 0.3 - 10 μ range, and less than 6000, 4500, or 3500 in the >0.5 μ range.

The fabrics of this invention have many uses, especially in the medical industry. Thus, in one embodiment the fabric is configured into a surgical fabric, preferably selected from the group consisting of gowns, drapes, and protective apparel. In another embodiment the fabric is configured into an absorbent pad, preferably selected from the group consisting of gauze, swabs, towels, and wipes. In still another embodiment the fabric is configured into an air filter.

The fabrics of this invention preferably derive their strength from the pressure liquid entanglement of fibers within the web. This is in contrast to poly(vinyl alcohol) fabrics of the prior art, in which the fabric derived its structural integrity and strength by weaving, binding adhesives, heat fusion, needlepunching, and stitchbonding. Thus, fabrics of the present invention are also distinct from prior art poly(vinyl alcohol) fabrics in several other respects, including one or more of the following: (1) the fabric is typically not woven to any substantial degree; (2) binding adhesives are typically absent from the fabric to any substantial degree; (3) the fabric is typically not heat fused to any substantial degree; (4) the fabric is typically not needlepunched to any substantial degree; and/or (5) the fabric is typically not stitchbonded to any substantial degree. "Substantial degree" refers to a level of bonding which contributes more to the strength of the fabric than entanglement from liquid jets.

Thus, the fabrics of this invention can also be characterized by properties other than the method of making described above. In a separate embodiment the invention provides a fabric in which (1) weaving is substantially absent, (2) binding adhesives are

substantially absent, (3) heat fusion is substantially absent, (4) needlepunching is substantially absent, and (5) stitchbonding is substantially absent. This distinct embodiment represents an alternative description of the fabrics of this invention, and it will be understood that the preferred process and fabric limitations discussed above 5 apply to this embodiment as well.

The process for making the fabrics of this invention also has several unique attributes which constitute separate embodiments of the described invention. Thus, in another embodiment the invention provides a method of finishing a poly(vinyl alcohol) fabric to impart water repellence to the fabric comprising contacting the fabric with an 10 aqueous finishing formulation, and subsequently drying the fabric and/or curing the finish at a temperature above the water solubility temperature of the poly(vinyl alcohol). In still another embodiment the invention provides a method of making a poly(vinyl alcohol) fabric comprising: (a) supporting a plurality of poly(vinyl alcohol) fibers on a mesh screen to form a web; (b) pressure liquid entangling the web; and (c) 15 drying the web. These distinct embodiments represent alternative descriptions of methods for producing the fabrics of this invention, and it will be understood that the preferred process and fabric limitations discussed above apply to these embodiments as well.

The following examples are put forth so as to provide those of ordinary skill in 20 the art with a complete disclosure and description of how the materials claimed herein are made and evaluated, and are intended to be purely exemplary of the invention and are not intended to limit the scope of what the inventors regard as their invention. Efforts have been made to ensure accuracy with respect to numbers (e.g., amounts, 25 temperature, etc.) but some errors and deviations should be accounted for. Unless indicated otherwise, parts are parts by weight, temperature is in °C or is at room temperature, and pressure is at or near atmospheric.

Example 1

The following is one set of processing data to make 65 gsm spunlace PVA fabric.

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Carding Feed Speed: 0.39 m/min
Feed Gap: 45.4 mm

Line Speed: 38.8 m/min
 Number of Layers: 6
 Web Width: 2323 mm
 Output Speed: 5.97 m/min
 5 Draw Ratio: 1.62
 Drawing Roller Speed: 6.6 8.0 9.3 10.7 m/min
 Water Pressure: 25 65 75 40 70 85 70 bar
 Speed : 10.3 10.5 11.2 m/min
 Drying Temperature: 120 °C
 10 Speed during drying: 11.4 m/min

Example 2 -- poly(vinyl alcohol) fabrics of the present invention

Test Item	Method	Fabric 1	Fabric 2	Fabric 3	Fabric 4
15	Weight oz/yd ² (gsm)	ASTM D3776-96 1.90 (64.5)	2.13 (72.8)	2.03 (68.9)	2.00 (67.9)
	Bursting strength (Mullen) (psi)	ASTM D3786-87 89.9	94.8	93.8	93.7
20	Air Permeability (Air flow)	ASTM D737-96 225.9 (CFM/sq. ft.)	202.8	202.7	188.2
	Tensile (Breaking) strength MD CD	ASTM D5035-95 (1" cut strip) 29.9	17.0 32.8	17.0 32.7	13.8 32.8
25	Elongation% MD CD	62.4 57.6	60.8 56.0	67.2 59.2	72.0 48.0
	Flammability FL0034 (RB)	16CFR1610(97) Class 1 (NF)	Class 1 (NF)	Class 1 (NF)	Class 1 (NF)
30	Original after D/C & Laun.	ASTM D1230-94 IBE DNI	DNI	DNI/IBE DNI	IBE DNI
	Mason jar saline repellency (min)	Isolyser 12-09 >90	>90	>90	>90

	Hydrostatic pressure (cm)	AATCC 127-95 (Suter)	32.2	30.3	33.5	33.8
5	Pilling resistance	ATSM D4970-98				
	Pilling (modified	@300 cycle	@300 cycle	@300 cycle	@450 cycle	
	Rupture (Martindale)	>4500	>4500	>4500	>4500	
	Face (Abrasion resistance)	Class 1	Class 1	Class 1	Class 1	
10	Back	Class 1	Class 1	Class 1	Class 1	
	Stiffness (cm)	ASTM D1388-96				
	Warp face	(Drapability)	3.2	3.1	2.85	2.30
	back		3.1	3.0	2.85	2.15
15	Filling face		2.7	3.0	2.85	2.70
	back		2.8	2.95	2.95	2.75
20	Impact penetration (IPR) (gms)	AATCC 42-94 (Water resistance)	0.38	0.53	0.54	1.44
	Water vapor transmission	ASTM E 96-95 (g/m ² /24 hrs)	1979.8	/	/	/
25	Linting	INDA IST				
	Cumulative	160.1-95				
	0.3-10μ	Total final count	3407	3299	1897	
30	>0.5μ	(CF)	2706	2906	1495	

Example 3 -- Comparative Fabrics

	Test Item	Method	FF (J & J) <u>-7555</u>	Sontara (Maxxim Medical) <u>-2321</u>
25	Weight oz/yd ² (gsm)	ASTM D3776-96	1.93 (65.6)	2.00 (67.9)
30	Bursting strength (Mullen) (psi)	ASTM D3786-87	34.3	24.6
	Air Permeability (Air flow)	ASTM D737-96 (CFM/sq. ft.)	75.7	63.7

	Tensile (Breaking) strength	ASTM D5035-95 (1" cut strip)	20.1	10.9
		CD (1b/in)	18.5	3.2
5	Elongation%	MD	24.0	30.4
		CD	27.2	59.2
	Flammability FL0034 (RB)	16CFR1610(97)	Class 1 (NF)	Class 1 (NF)
10	Original after D/C & Laun.	ASTM D1230-94	7.6s BB 5.9s BB	10.1s BB 6.0s BB
	Mason jar saline repellency (min)	Isolyser 12-09	>90	>90
	Hydrostatic pressure (cm)	AATCC 127-95 (Suter)	32.8	33.0
15	Pilling resistance	ATSM D4970-98		
	Pilling	(modified Martindale)	@200	@1300*
	Rupture	(Abrasion resistance)	@2500	@2800
	Face		Class 3**	Class 1***
	Back		Class 1	Class 1
20	Stiffness (cm)	ASTM D1388-96		
	Warp face	(Drapability)	2.6	2.85
	back		3.55	3.15
	Filling face		1.7	1.90
	back		1.6	1.80
25	Impact penetration (IPR) (gms)	AATCC 42-94 (Water resistance)	13.68	1.05
	Water vapor transmission	ASTM E 96-95 (g/m ² /24 hrs)	/	1609.4
30	Linting	INDA IST		
	Cumulative	160.1-95		
	0.3-10 μ	Total final count	7799	20763
	>0.5 μ	(CF)	6668	17895

"RB": Rate of Burning (sec) on 45° Flammability Tester

35 "NF": Normal Flammability

"DNI": Did Not Ignite

"IBE": Ignited But Extinguished

"BB": Time in seconds, Base Burn

"FR": Flexural Rigidity

"Class 1(NF)" in Flammability: Passes the requirement of 16 CFR 1610 as "Class 1: Normal Flammability"

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"@1300 cycle*" in Pilling: Fuzzing observed at 300 cycles

Pilling test condition: 9 kpa load, abradant is plain weave cross bred and worsted wool fibers, 5,000 cycles maximum.

"Class 1" in Pilling:: very severe pilling

10 "Class 3**" in Pilling: moderate pilling but severe fabric rupture

"Class 1***" in Pilling: Very severe fabric rupture

"> 90" in Mason Jar: Fluid penetration has not occurred within 90 min.

"CF": Particulate per Cubic Foot of sampled air per specimen

15 Throughout this application, various publications are referenced. The disclosures of these publications in their entireties are hereby incorporated by reference into this application in order to more fully describe the state of the art to which this invention pertains.

It will be apparent to those skilled in the art that various modifications and
20 variations can be made in the present invention without departing from the scope or spirit of the invention. Other embodiments of the invention will be apparent to those skilled in the art from consideration of the specification and practice of the invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with a true scope and spirit of the invention being indicated by the
25 following claims.